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Gas Sample Preparation and Calibration of Thermal Conductivity Gas Analysers

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ABSTRACT

This report describes the sample gas preparation technique and calibration procedure for thermal conductivity gas analysers operated by the Platform Sciences Laboratory. The analysers are used to measure gaseous fire suppressant concentrations of extinguishing systems installed in Royal Australian Navy ships. These measurements are undertaken to ensure the systems meet the minimum standards for acceptance into service.

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Gas Sample Preparation and Calibration of Thermal Conductivity Gas Analysers

Executive Summary

Thermal conductivity analysers are a means of measuring gas concentration and have a role in fire suppressant concentration measurements during system validation assessments.

This report describes the calibration gas sample preparation and the calibration procedure for thermal conductivity gas analysers operated by the Platform Sciences Laboratory.

The primary purpose of these analysers is to measure the concentration of fire extinguishment gases in protected spaces onboard vessels of the Royal Australian Navy. These measurements are usually conducted prior to handover to ensure that the system output meets the specified requirements.

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1. Introduction

Fire fighting suppressant gases are used to protect Defence platforms including ships, aeroplanes, land vehicles and shore facilities. There are two reasons for fitting fire protection equipment in these platforms and buildings (i) to protect the asset in the event of a fire and (ii) to protect personnel. For gaseous suppressants to be effective they must be activated and discharged in a prescribed period of time and maintain the extinguishing concentration for another prescribed period. To validate these requirements, the gas concentration is measured during a test release of the particular extinguishant. These measurements can be undertaken using thermal conductivity analysers where gas concentration is inferred from measures of thermal conductivity. The result is a concentration history from which the acceptability of the extinguishing system can be assessed.

The Platform Sciences Laboratory has acquired 5 analysers (see Figure 1), each having 6 measurement channels providing the capability to comprehensively characterise fire extinguishant gas distribution in large spaces such as ship machinery rooms. The analysers were manufactured by Tripoint Instruments, USA.

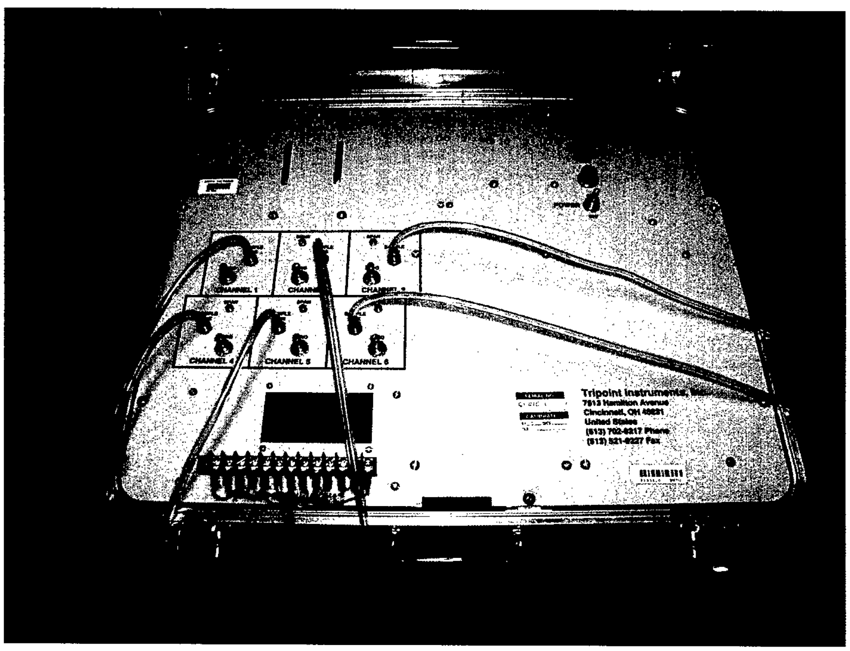


Figure 1 Gas Analyser unit with six channels

2. Description of analyser

The gas analysers will measure the volume concentration of gases in air by determining the thermal conductivity of a particular gas sample and comparing that value to a calibration curve derived from the analyser response over a range of gas concentrations.

2.1 Operation

Differences in the thermal conductivity of gases make it possible to determine the concentration of one gas in another by measuring the thermal conductivity of the gas mixture and comparing it with a reference value.

The analysers are based on thermal conductivity cells, which have an electrical filament inside a closed volume. Each analyser comprises two cells, the measurement cell and the reference cell. When a voltage is applied to a filament, current flow causes it to heat up resulting in a change in resistance. The increase in temperature will be dependent on the rate of heating of the filament due to the applied voltage and the rate of cooling due to the conductivity of the gas surrounding the filament. An equilibrium temperature will be reached from which the gas concentration can be inferred.

When the sample gas and the reference gas are identical, the filament resistances are matched and the gas concentration being measured is the same as the gas concentration in the reference cell. When a different concentration sample gas is fed into the measurement cell, the gas flow initially cools the filament however, when the flow stops, the temperature in the measurement cell will increase to an equilibrium value that is dependant on the gas concentration. The only variable between the measurement and reference cells is thermal conductivity of the gas surrounding the filament. The change in resistance produces a measurable voltage change, which can be equated with the gas concentration.

2.2 Voltage output

The output voltage for each channel can be adjusted depending on the sensitivity required.

2.3 Measurement cycle

A fresh batch of sample gas is drawn into the measurement cell every four seconds. The sample is drawn into the measurement cell and isolated via a solenoid valve after 2 seconds allowing the cell to be purged of any gas sample from the previous reading and bring the temperature back to the an initialising temperature. During the remainder of the cycle, the filament temperature reaches an equilibrium condition from which a measure of the gas concentration can be made. The output from the measurement cell is monitored continuously but should only be read at the end of the 4-second cycle.

2.4 Conductivity cell linearity

The analyser response is linear to +/-2% of full scale output (50mV).

3. Calibration

The calibration procedure for the gas analysers involves preparing various volume percent mixtures of the sample gas in air and producing a calibration curve from which the responses to unknown gas concentrations are compared. These sample gas mixtures are drawn into the measurement cells in the analyser via a vacuum pump. The air and sample gas volumes required to make up the specific concentrations are measured using syringes and injected into a valved sample bag.

3.1 Procedure

The gas mixtures are prepared in ultra low permeability flexible gas sample bags that are suitably valved to prevent gas leakage during filling or testing. A 5 litre bag is shown in Figure 2. The sample bags are evacuated prior to use.

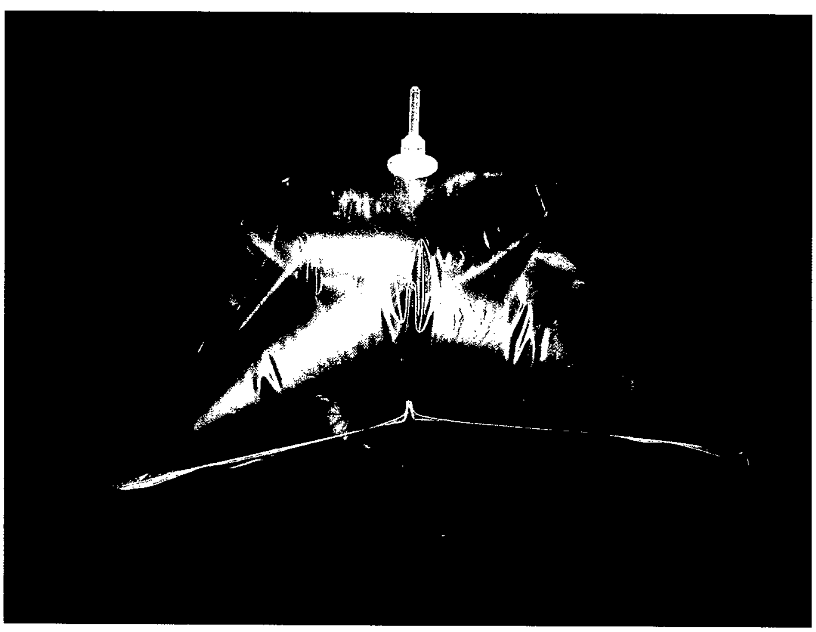


Figure 2 Gas sample bag

The large bag volume ensures a sufficient supply of gas to calibrate all the measurement channels at a particular concentration and minimise variations in concentration that may occur between a large number of smaller gas batches. The sample gas/air mixture is introduced into the bag via syringes. A two-litre syringe is used for measuring volumes of air and a smaller 100ml syringe to measure the sample gas. These syringes are shown in Figure 3

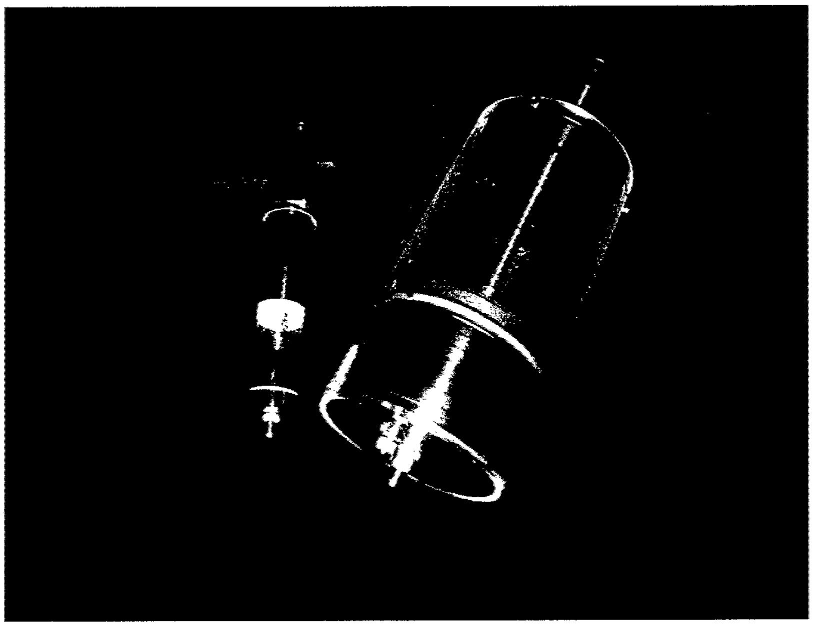


Figure 3 Syringes for measuring volumes of air and sample gas

Prior to filling a bag with air or gas, the bag volume is flushed with air and evacuated a number of times to eliminate contamination from previous sampling.

Air is drawn into the larger of the two syringes; the syringe is open to the atmosphere i.e. at atmospheric pressure, therefore the volume of air within the syringe is the volume marked on the syringe. A short connecting tube is placed between the outlet end of the syringe and the valve on the bag and the contents of the syringe discharged into the sample bag, see Figure 4. The contents of the syringe are discharged into the sample bag and any volume of air contained within the connecting tube prior to discharge remains in the tube when the sample bag valve is closed. This procedure results in a precise volume of air discharged into the sample bag.

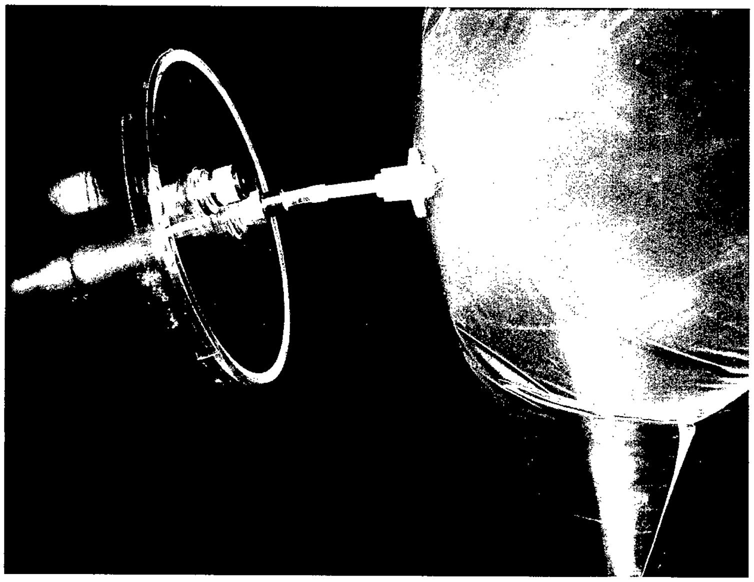


Figure 4 Air discharge into the gas sample bag

The sample gas is supplied in a pressurised cylinder and transferred to the smaller syringe by this pressure, see Figure 5. A small length of tube is connected to the gas cylinder which is then opened to produce a gas flow to purge any air in the tubing. The syringe is connected to the tubing and the gas released into the syringe, the pressure exerted by the gas forcing the syringe plunger open and filling it with the sample gas. The syringe is over filled, so that any air discharged into the syringe will be forced to the top by the denser sample gas, which is expelled by closing the plunger by hand. This process also occurs at atmospheric pressure so the volume of sample gas in the syringe is additive with the volume of air in the sample bag. This process should be carried out with the syringe outlet positioned vertically upright to minimise any gas loss. A short connector piece is used to join the syringe and the sample bag and the gas is discharged into the sample bag. The discharge is carried out with the syringe pointed downward so that all of the sample gas is emptied into the bag.

Figure 5. Transfer of FM200 from pressurised cylinder to syringe

The gas must be thoroughly mixed prior to the concentration measurements to ensure that a constant concentration of the sample gas is drawn into the analyser. This requires turbulent flow within the bag and is achieved by rotating the sample bag at a moderate to fast rate at least 10 times.

3.2 Example of calibration of analysers

The analysers were calibrated for the fire extinguishant FM200 although the procedure can be used for any gas. FM200 also known as HFC-227ea is a hydrofluorocarbon with the chemical name heptafluoropropane and formula CF_3-CFH-CF_3 and supplied as 99.9% minimum purity.

Gas/air mixtures of 0%, 5%, 10% and 15% FM200 were chosen for the calibration range, 15% represents the upper limit of FM200 concentration and four readings including the zero reading give a spread of data that should show irregularities such as non-linearity. Sample gas volumes of 5 litres were prepared with sample gas/air volumes listed in Table 1.

The analysers are equipped with a span control that can vary the sensitivity at each channel individually. The span levels were not altered during the calibration.

Table 1 Volumes of air and FM200 to produce the 5, 10 and 15% FM200 concentrations

Total gas volume (ml)	%FM200	Volume of air (ml)	Volume of FM200 (ml)
5000	0	5000	0
	5	4750	250
	10	4500	500
	15	4250	750

3.3 Measurement uncertainties

3.3.1 Uncertainty in gas concentration volumes

The syringes used to measure the gas volumes have a finite capacity and require multiple fills to produce the required volume of gas for testing, compounding the measurement uncertainty. The syringes also have resolution limits, the large syringe is graduated in 100ml divisions but can be read reliably to 25ml, the smaller syringe has a resolution of 1ml. The cumulative precision of multiple gas fills is the square root of the sum of the squares of the individual uncertainties.

A 5 litre bag of each of the gas concentrations will result in the following measurement uncertainties

5% gas concentration

Volume of air	4750ml
Volume of syringe	2000ml
Number of measures	3
Reading precision	+/-25ml
Cumulative precision error	$\sqrt{1875}$ml
Measurement and error	4750+/-43ml

Volume of FM200	250ml
Volume of syringe	100ml
Number of measures	3
Instrument precision	1ml
Cumulative precision error	$\sqrt{3}$ml
Measurement and error	250+/-2ml

The maximum and minimum total gas volumes possible for a 5% FM200 gas concentration are
4707ml+248ml = 4955ml
4707ml+252ml = 4959ml

4793ml+248ml = 5041ml
4793ml+252ml = 5045ml

and the possible gas concentrations

248/4955 * 100 = 5.0% FM200
252/4959* 100 = 5.1% FM200
248/5041 * 100 = 4.9% FM200
252/5045 * 100 = 5.0% FM200

For a nominal 5% FM200 gas concentration, the maximum measurement error is +/-0.1% FM200.

10% gas concentration

Volume of air measured	4500ml
Volume of syringe	2000ml
Number of measures	3
Reading precision	+/-25ml
Cumulative precision error	√1875ml
Measurement and error	4500+/-43ml

Volume of FM200 measured	500ml
Volume of syringe	100ml
Number of measures	5
Instrument precision	1ml
Cumulative precision error	√5ml
Measurement and error	500+/-2ml

The maximum and minimum total gas volumes possible for a 10% FM200 gas concentration are

4457ml+498ml = 4955ml
4457ml+502ml = 4959ml
4543ml+498ml = 5041ml
4543ml+502ml = 5045ml

and the possible gas concentrations are

498/4955 * 100 = 10.0% FM200
502/4959 * 100 = 10.1% FM200
498/5041 * 100 = 9.9% FM200
505/5045 * 100 = 10.0% FM200

For a nominal 10% FM200 gas concentration, the maximum measurement error is +/- 0.1 FM200

15% gas concentration

Volume of air measured	4250ml
Volume of syringe	2000ml
Number of measures	3
Reading precision	25ml
Cumulative precision error	$\sqrt{1875}$ml
Measurement and error	4250+/-43ml

Volume of FM200 measured	750ml
Volume of syringe	100ml
Number of measures	8
Instrument precision	1ml
Cumulative precision error	$\sqrt{7}$ml
Measurement and error	750+/-3ml

The maximum and minimum total gas volumes possible for a 15% FM200 gas concentration are

4207+747ml = 4954ml
4207+753ml = 4960ml
4293+747ml = 5040ml
4293+753ml = 5046ml

and the possible gas concentrations are

747/4954 * 100 = 15.1% FM200
753/4960 * 100 = 15.2% FM200
747/5040 * 100 = 14.8% FM200
753/5046 * 100 = 14.9% FM200

For a nominal 15% FM200 gas concentration, the maximum measurement error is +/-0.2% FM200.

3.3.1 Analyser output uncertainty

The output from each analyser cell is acquired and stored on a PC for data analysis at a latter date. The data is stored as data pairs where one of the data pair represents time and the other, the analyser response. Figure 6 shows a typical response from one of the gas analysers.

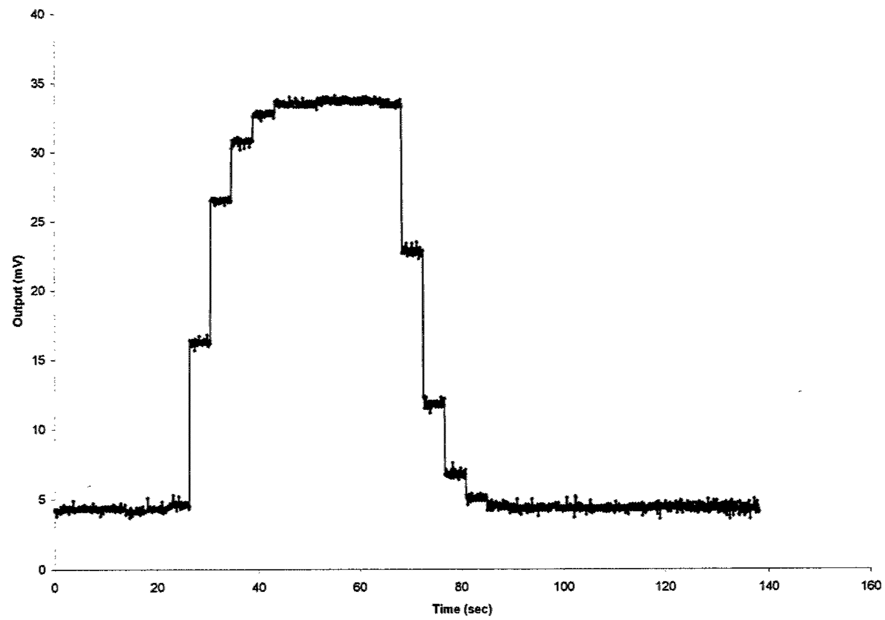


Figure 6 Typical gas analyser output

Data analysis comprised smoothing the data to remove unwanted noise, determining and applying a zero offset and measuring the peak response for a particular gas concentration. The outputs for gas concentrations of 0%, 5%, 10% and 15% FM200 were measured for each channel and plotted to produce calibration graphs. The calibration data are shown in Tables 2-7 and the corresponding calibration graphs in Figures 7-11.

The uncertainty in the output (mV) data was determined by subjecting the data to a statistical analysis. One standard deviation of the output was taken as the uncertainty in the measurement; the probability that the output is within +/- one standard deviation is approximately 68%. The uncertainty in the gas concentration measurement was determined from the volume measurement uncertainties. The uncertainties are presented as error bars on the calibration graphs.

Table 2 Calibration responses for Box 1

Box No.	Channel No.	%FM200	Output (mV)
1	1	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 6.3 +/- 0.3 14.1 +/- 0.3 23.9 +/- 0.3
1	2	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 9.4 +/- 0.4 17.0 +/- 0.4 25.7 +/- 0.4
1	3	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 9.2+/- 0.2 16.6 +/- 0.2 24.7 +/- 0.2
1	4	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 8.6 +/- 0.2 15.1 +/- 0.2 22.9 +/- 0.3
1	5	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 9.6 +/- 0.2 17.0 +/- 0.2 25.7 +/- 0.2
1	6	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 8.9 +/- 0.1 15.6 +/- 0.2 23.6 +/- 0.2

Table 3 Calibration responses for Box 2

Box No.	Channel No.	%FM200	Output (mV)
2	1	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.3 +/- 0.1 18.6 +/- 0.1 27.7 +/- 0.2
2	2	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.3 +/- 0.1 18.2 +/- 0.2 27.2 +/- 0.2
2	3	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 9.5 +/- 0.5 17.1 +/- 0.5 25.3 +/- 0.5
2	4	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.1 +/- 0.3 17.9 +/- 0.3 27.1 +/- 0.3
2	5	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 9.6 +/- 0.2 16.6 +/- 0.3 24.1 +/- 0.3
2	6	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 9.7 +/- 0.2 16.7 +/- 0.2 25.7 +/- 0.2

Table 4 Calibration responses for Box 3

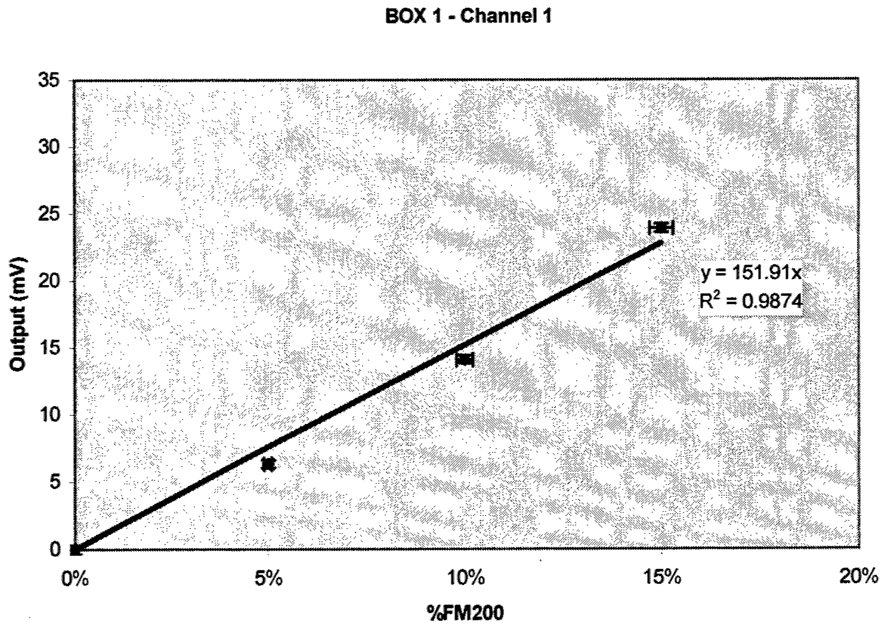
Box No.	Channel No.	%FM200	Output (mV)
3	1	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.8+/- 0.1 20.7 +/- 0.1 29.1 +/- 0.2
3	2	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.4+/- 0.1 21.5 +/- 0.1 30.3 +/- 0.1
3	3	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.2 +/- 0.1 21.0 +/- 0.2 30.0 +/- 0.2
3	4	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.4 +/- 0.1 21.6 +/- 0.1 30.6 +/- 0.1
3	5	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.2 +/- 0.1 21.1 +/- 0.1 29.0 +/- 0.3
3	6	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.0 +/- 0.2 20.7 +/- 0.2 29.6 +/- 0.2

Table 5 Calibration responses for Box 4

Box No.	Channel No.	%FM200	Output (mV)
4	1	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.1 +/- 0.3 23.0 +/- 0.3 31.6 +/- 0.3
4	2	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.0 +/- 0.1 21.2 +/- 0.2 29.4 +/- 0.2
4	3	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.7 +/- 0.1 22.2 +/- 0.1 30.7 +/- 0.1
4	4	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.9 +/- 0.1 21.0 +/- 0.2 28.7 +/- 0.1
4	5	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.7 +/- 0.2 22.6 +/- 0.2 31.2 +/- 0.2
4	6	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.2 +/- 0.2 21.4 +/- 0.2 29.4 +/- 0.2

Table 6 Calibration responses for Box 5

Box No.	Channel No.	%FM200	Output (mV)
5	1	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.0 +/- 0.2 21.4 +/- 0.2 30.3 +/- 0.2
5	2	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.4 +/- 0.2 22.0 +/- 0.2 30.8 +/- 0.3
5	3	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.7 +/- 0.2 21.3 +/- 0.2 29.4 +/- 0.3
5	4	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.5 +/- 0.1 21.1 +/- 0.1 30.2 +/- 0.1
5	5	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 10.8 +/- 0.1 21.1 +/- 0.1 29.8 +/- 0.1
5	6	0 5.0 +/- 0.1 10.0 +/- 0.1 15.0 +/- 0.2	0 11.2 +/- 0.1 21.7 +/- 0.1 30.6 +/- 0.1



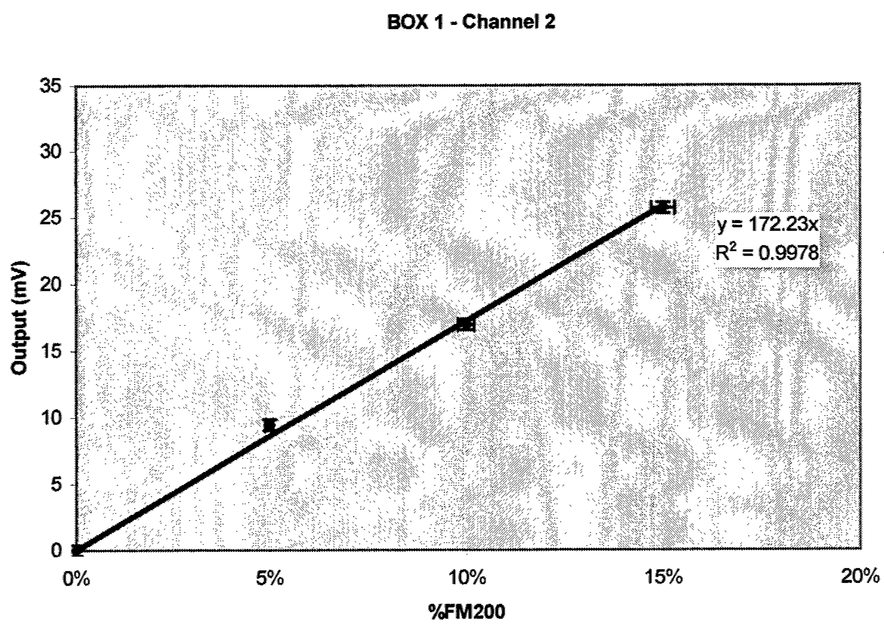
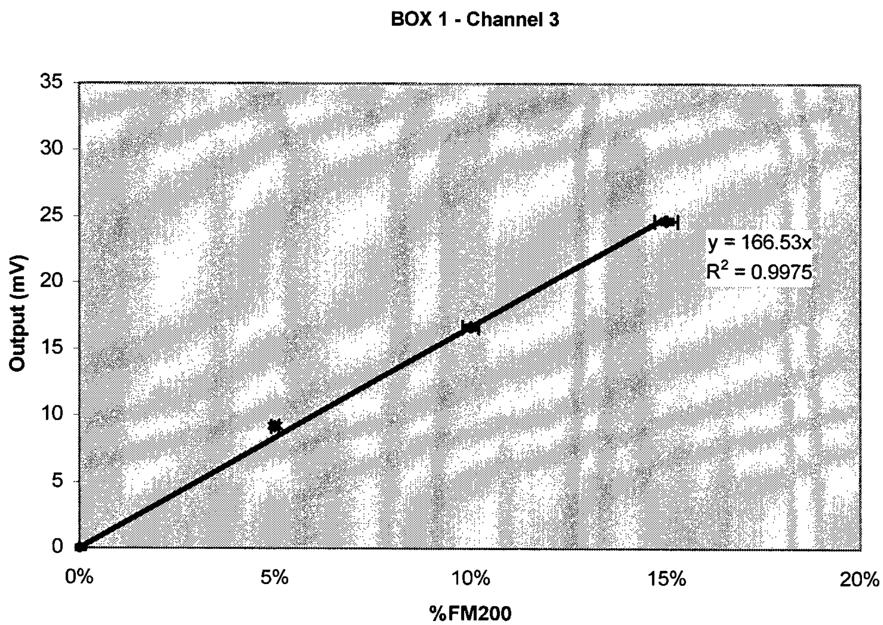
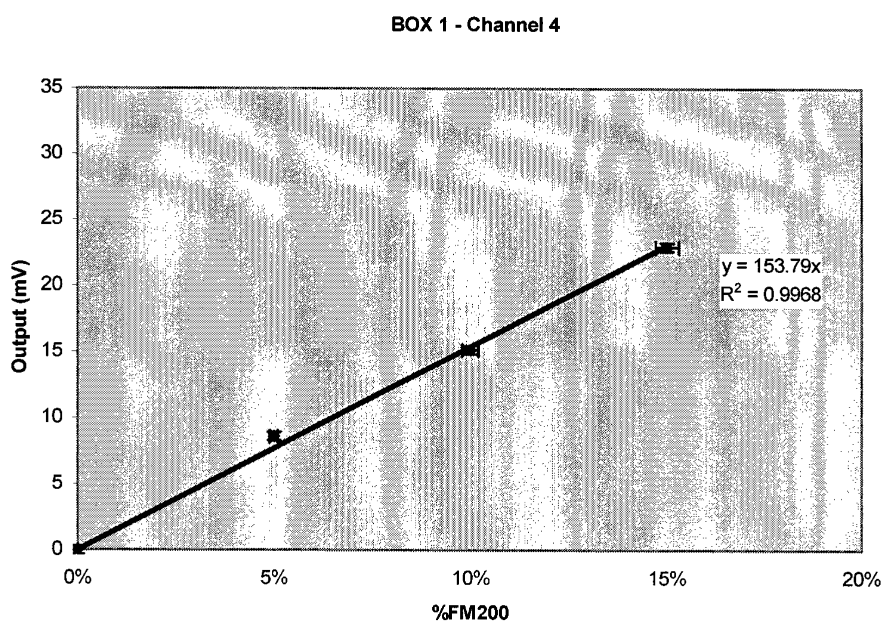


Figure 7 Calibration graphs for Box 1





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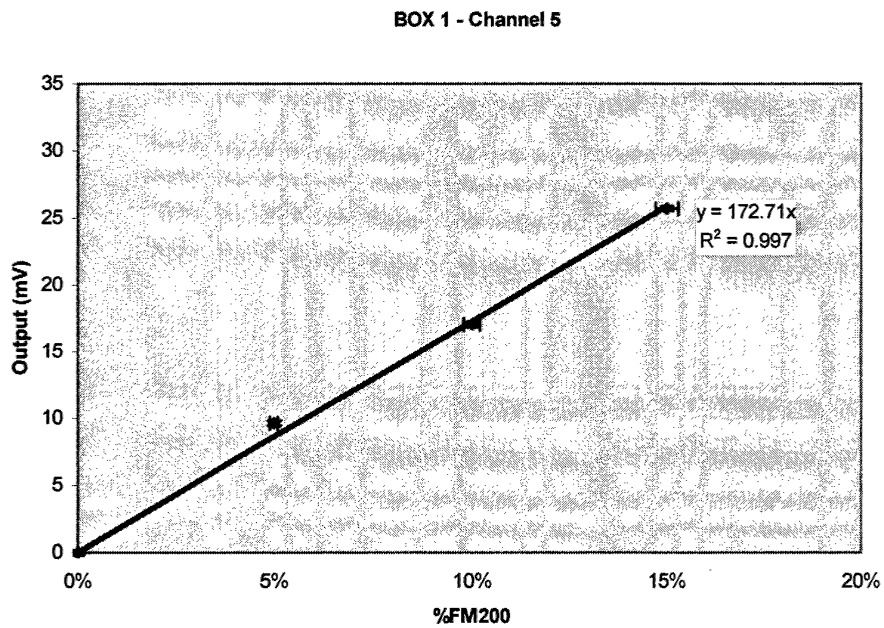

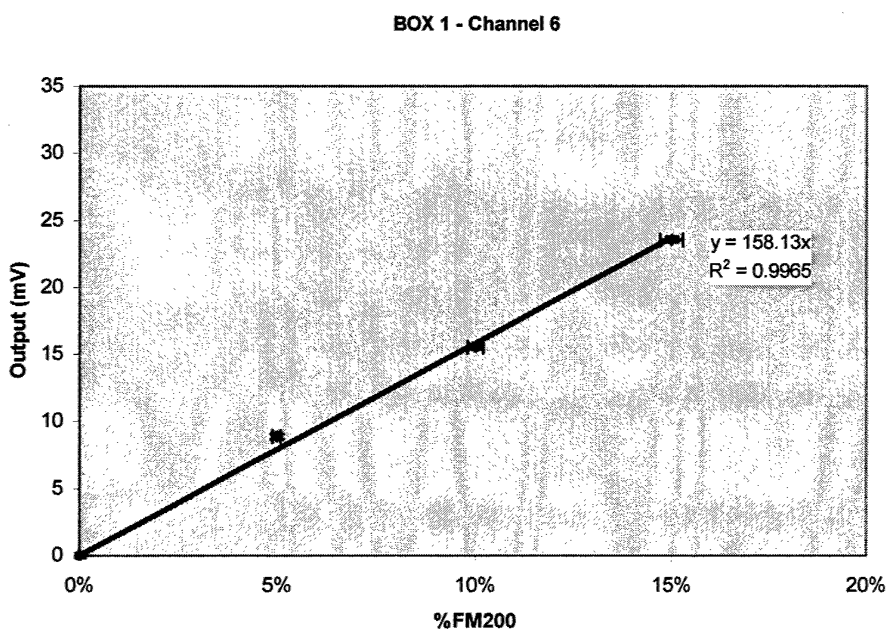

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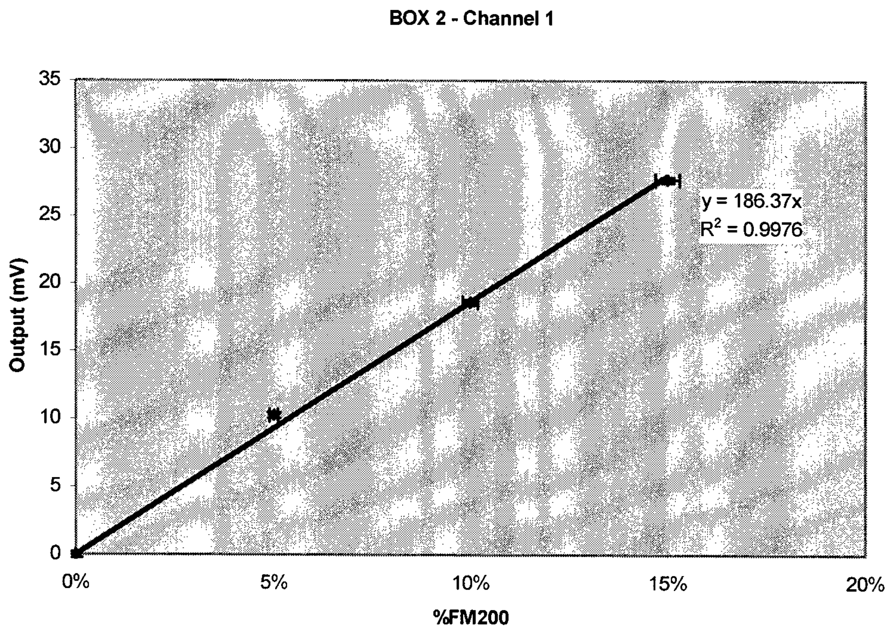


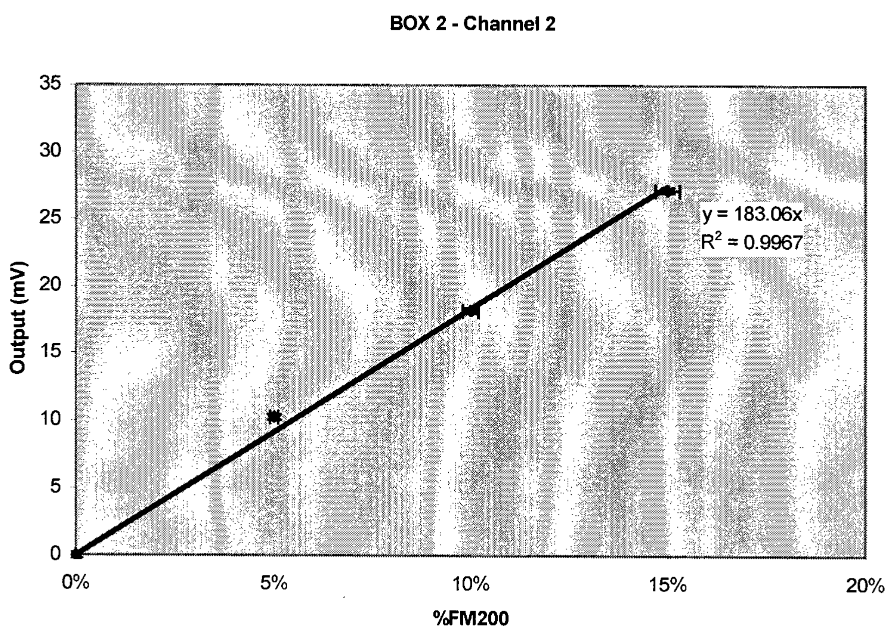
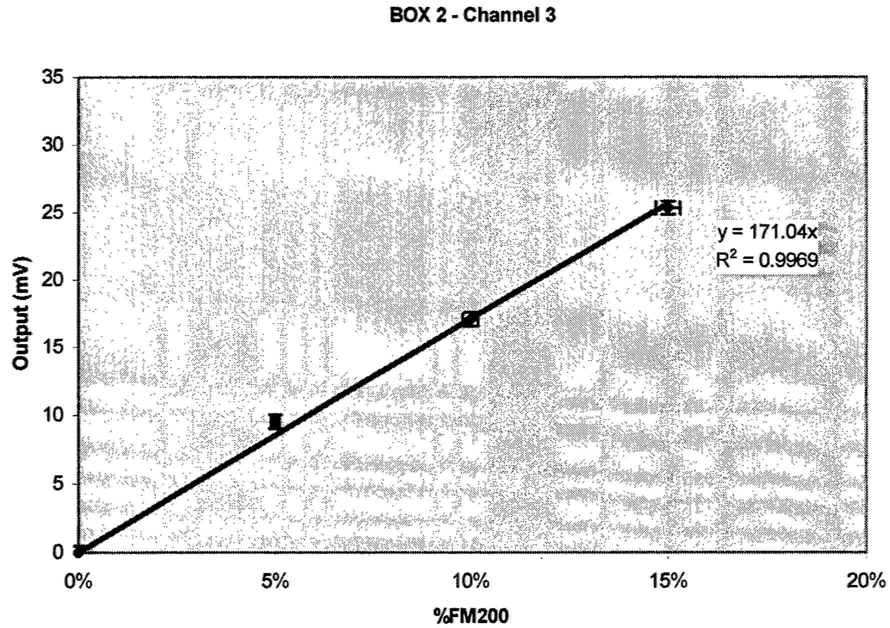
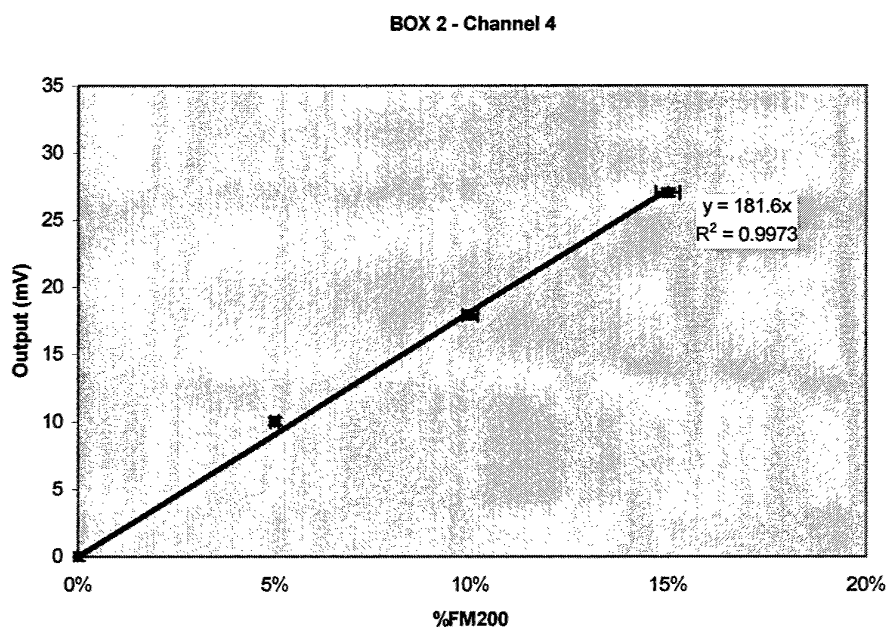


Figure 8 Calibration graphs for Box 2





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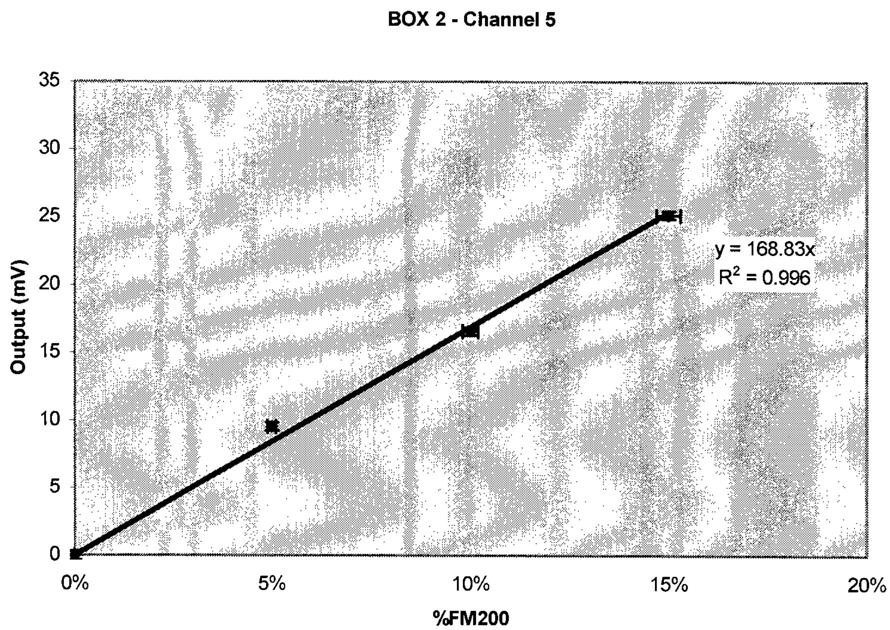

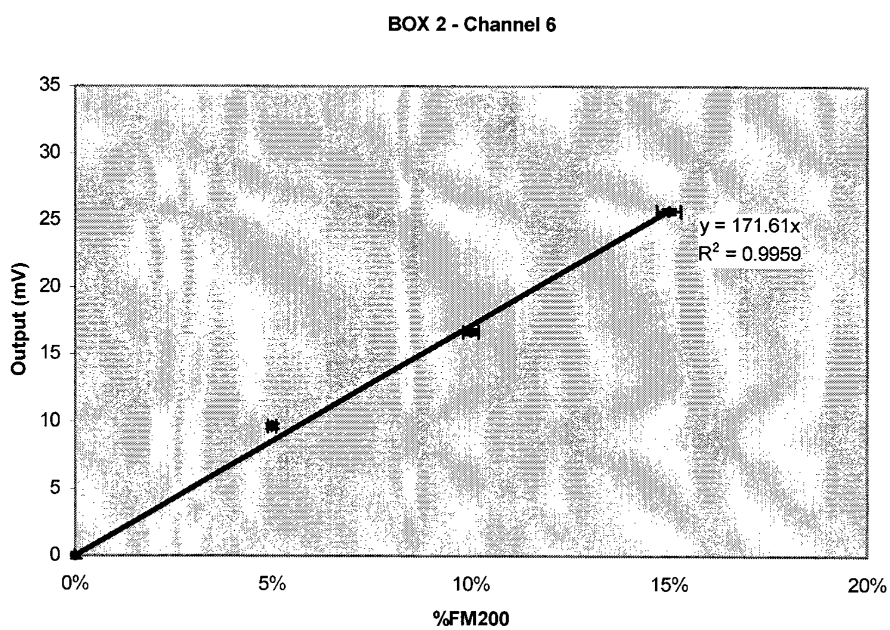

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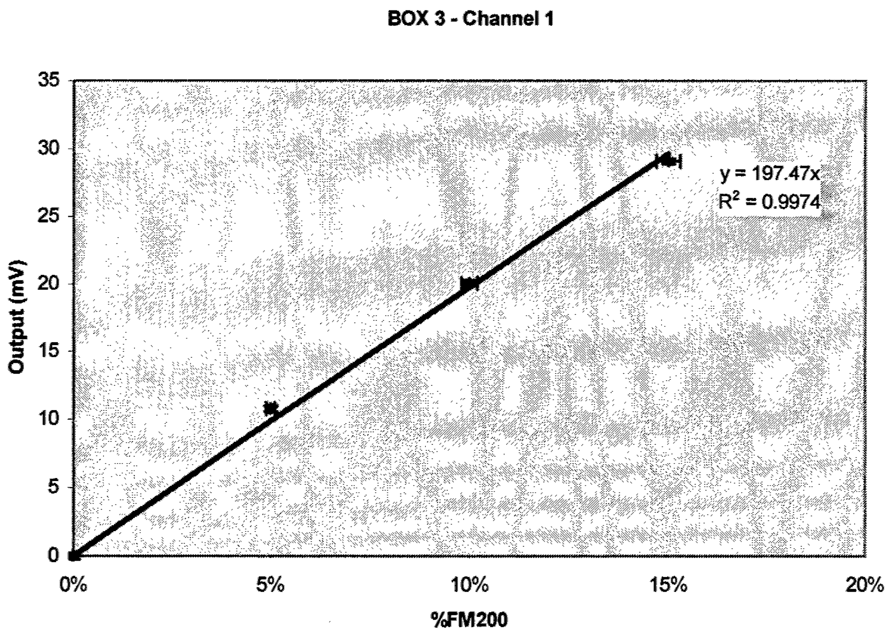


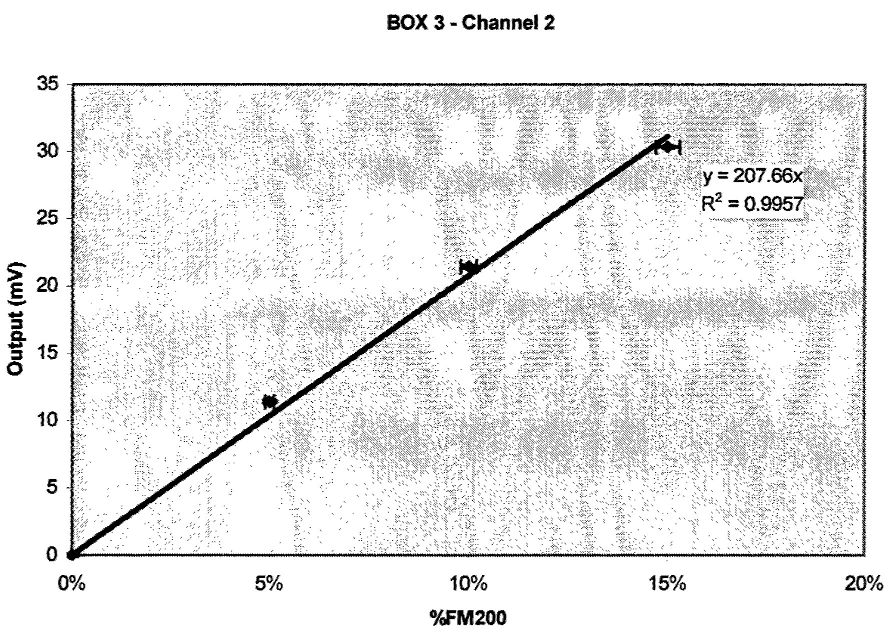
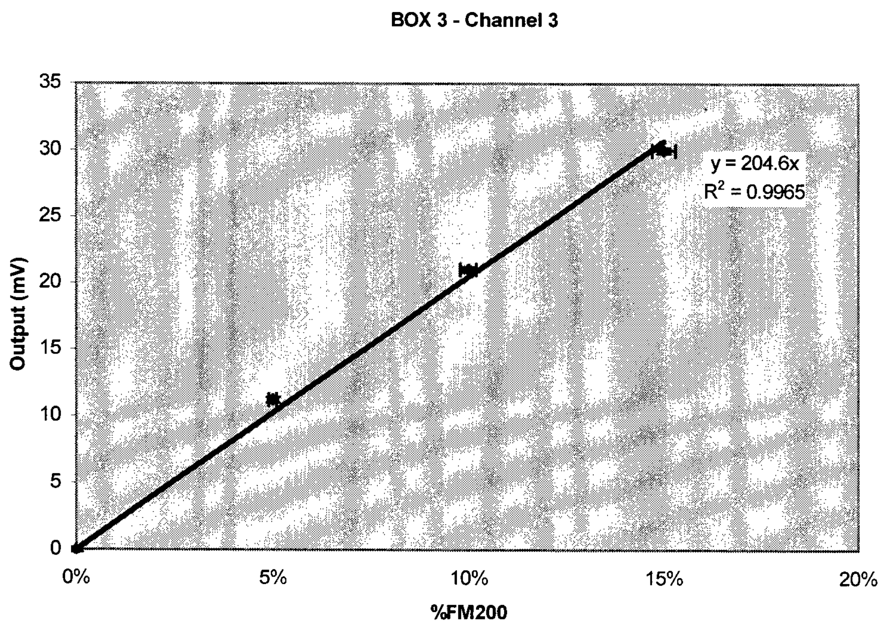


Figure 9 Calibration graphs for Box 3



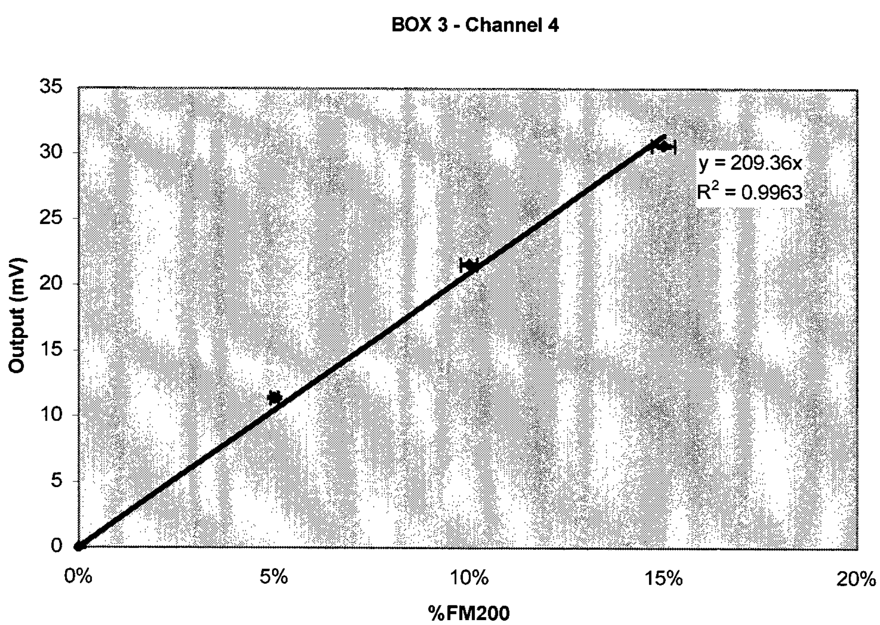
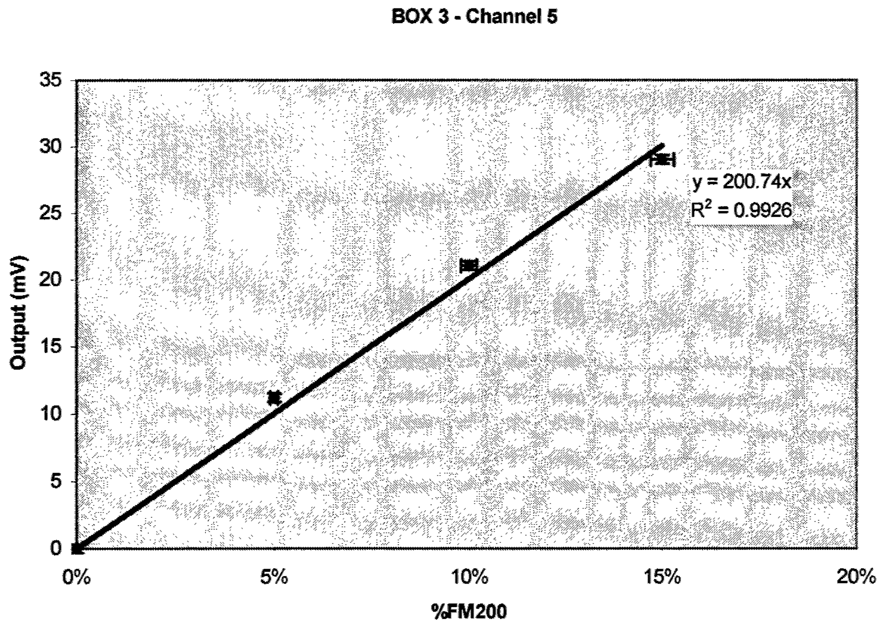


Figure 9(continued) *Calibration graphs for Box 3*



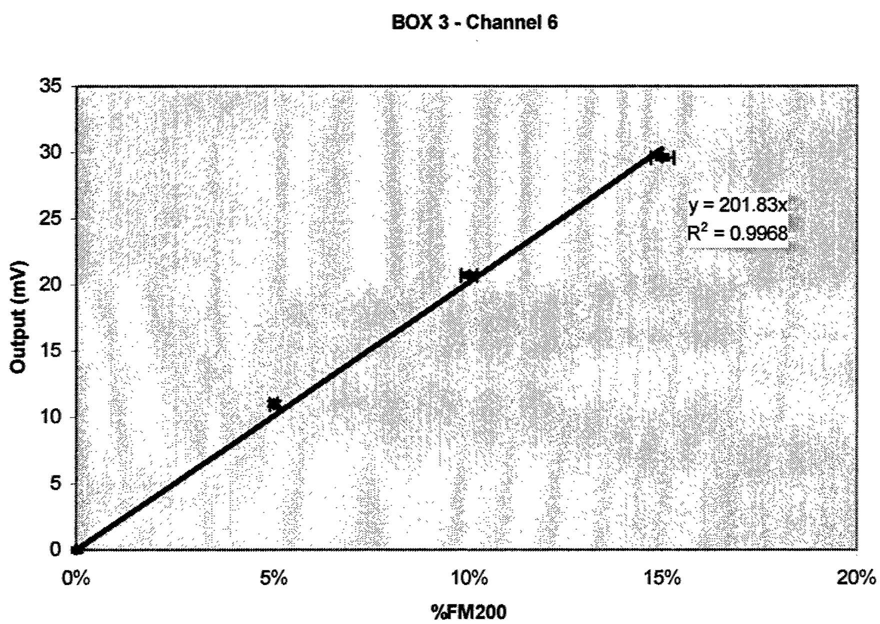
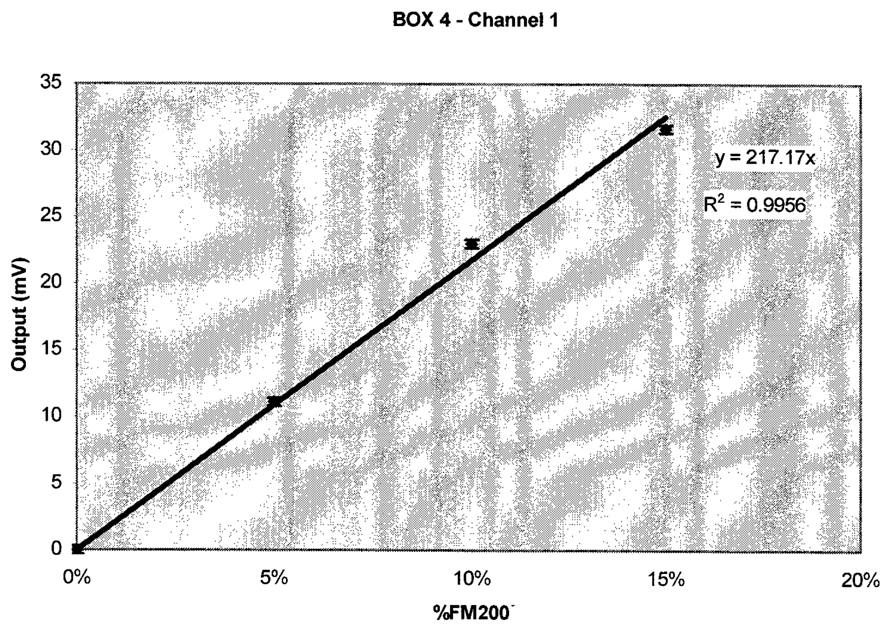


Figure 9(continued) Calibration graphs for Box 3



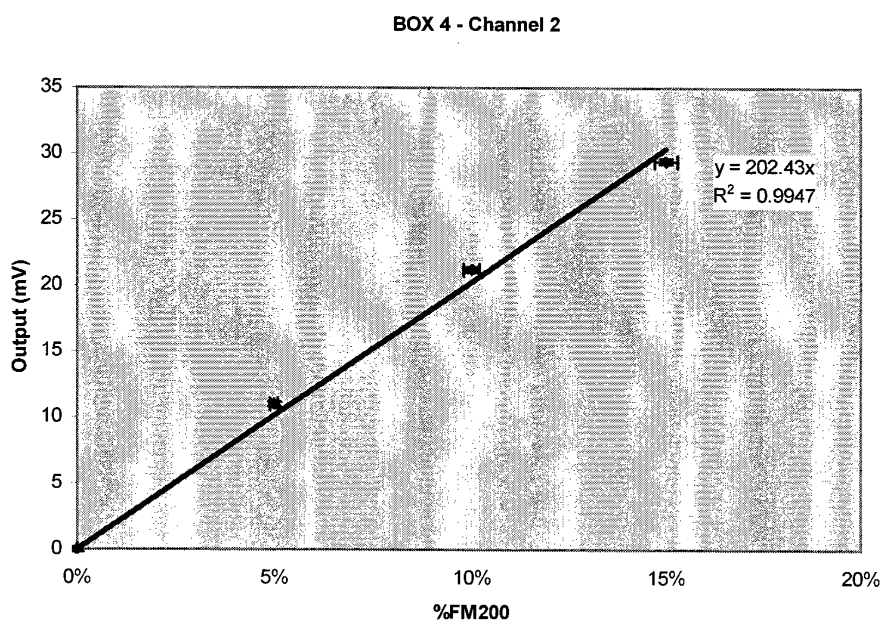
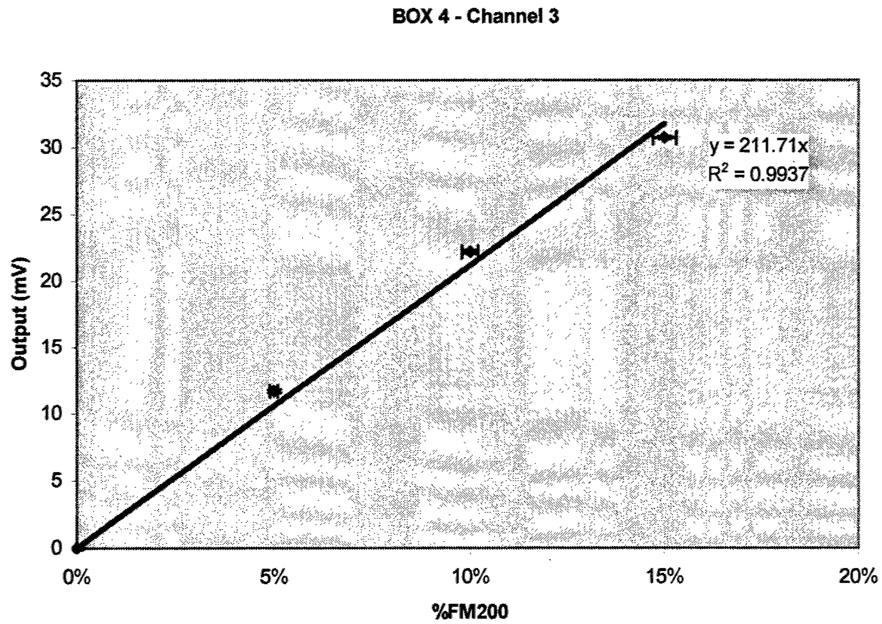


Figure 10 Calibration graphs for Box 4



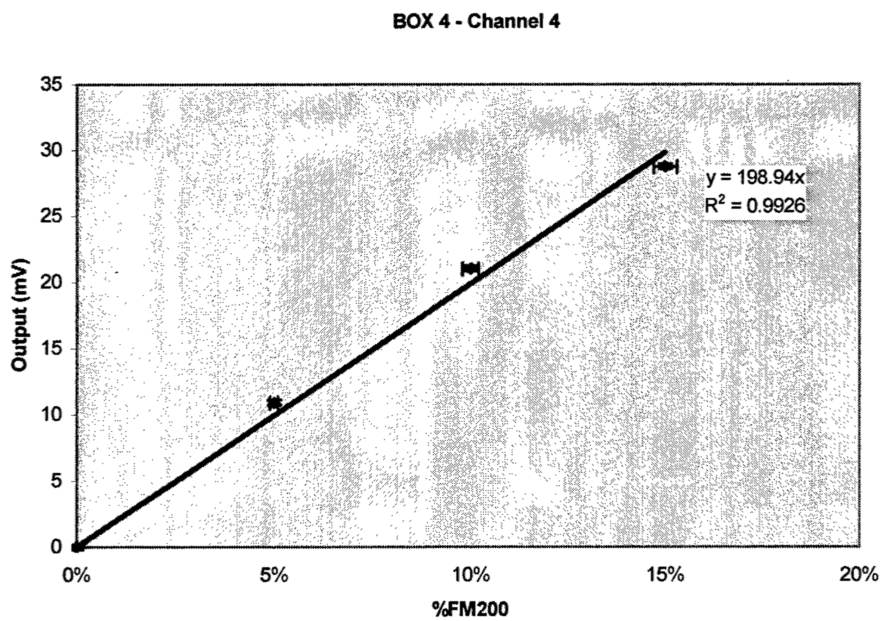
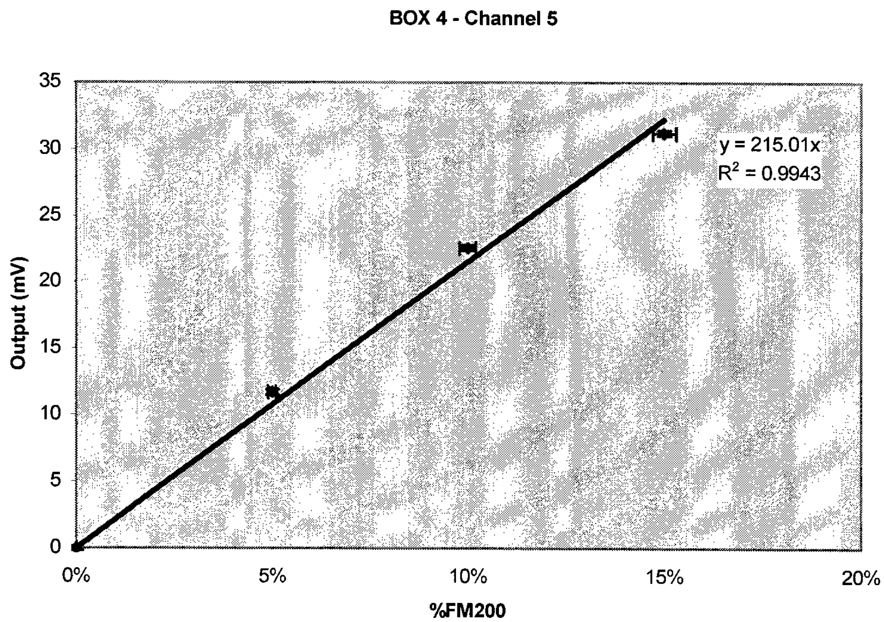


Figure 10(continued)   Calibration graphs for Box 4



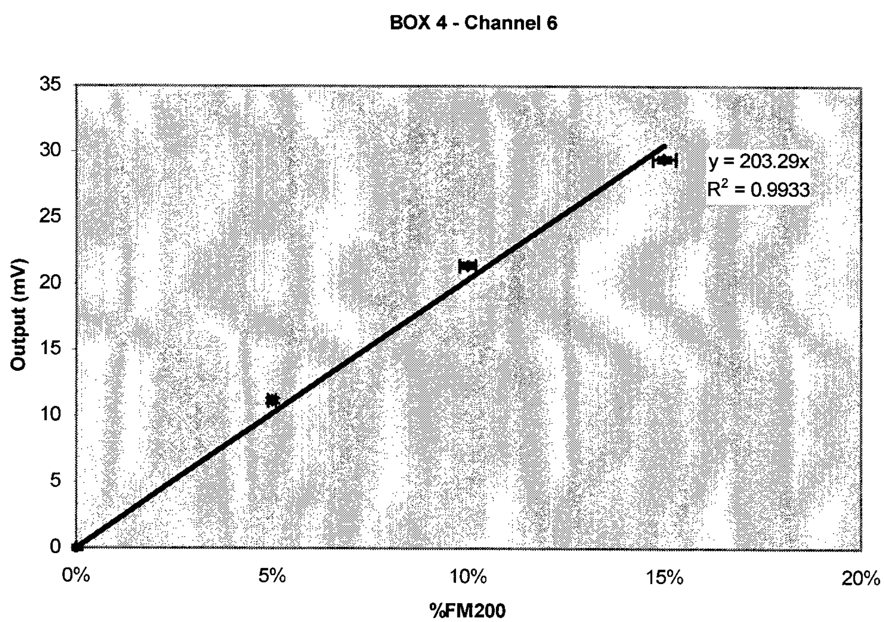
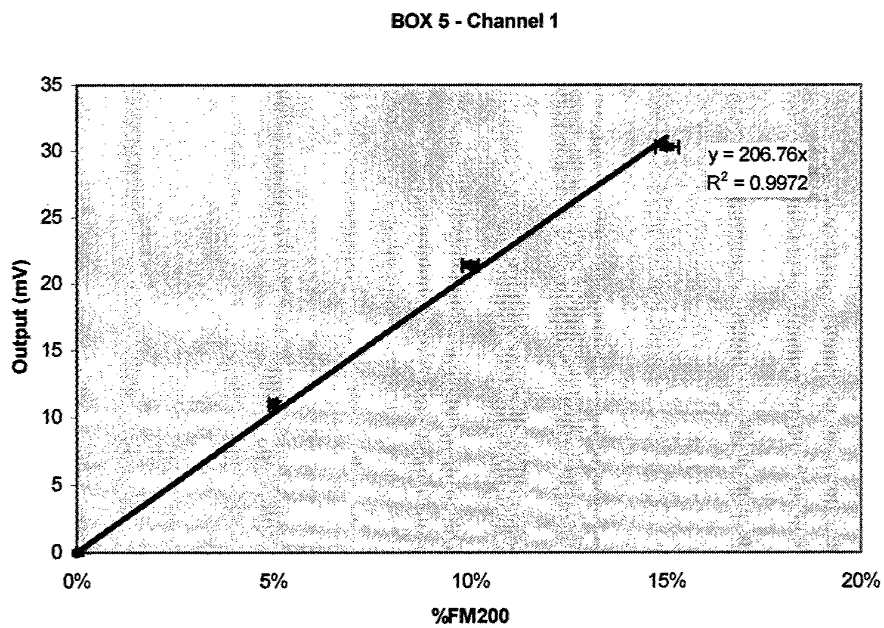


Figure 10(continued) Calibration graphs for Box 4



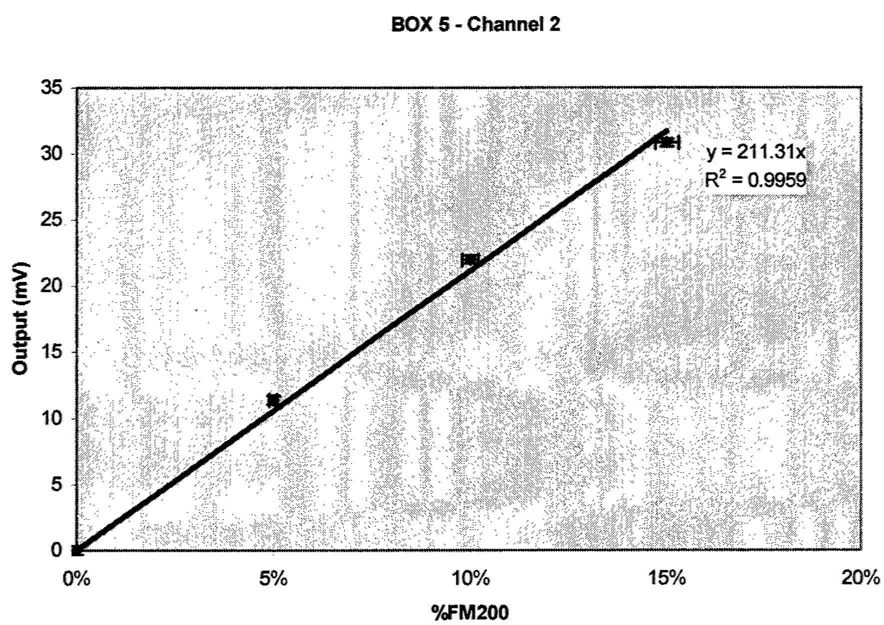
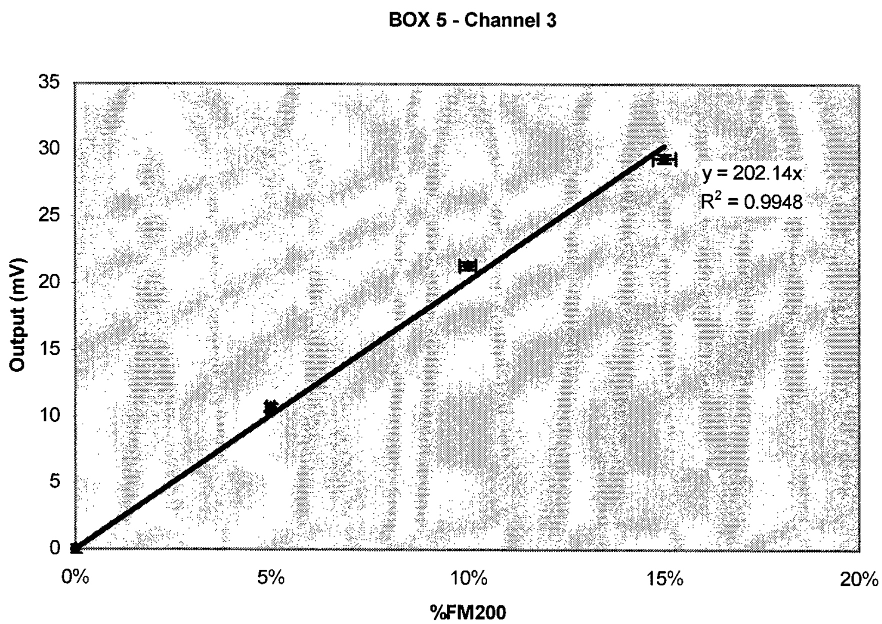


Figure 11 Calibration graphs for Box 5



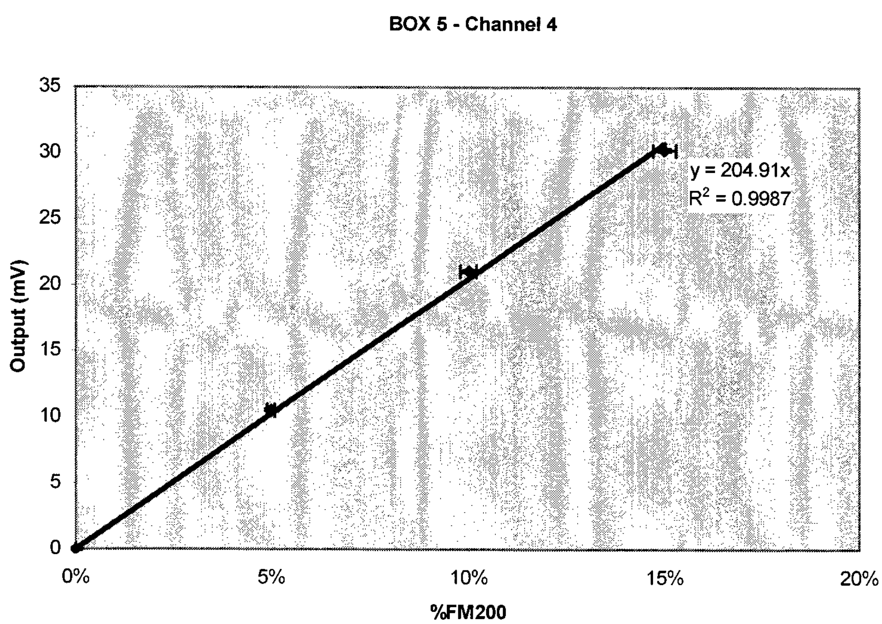
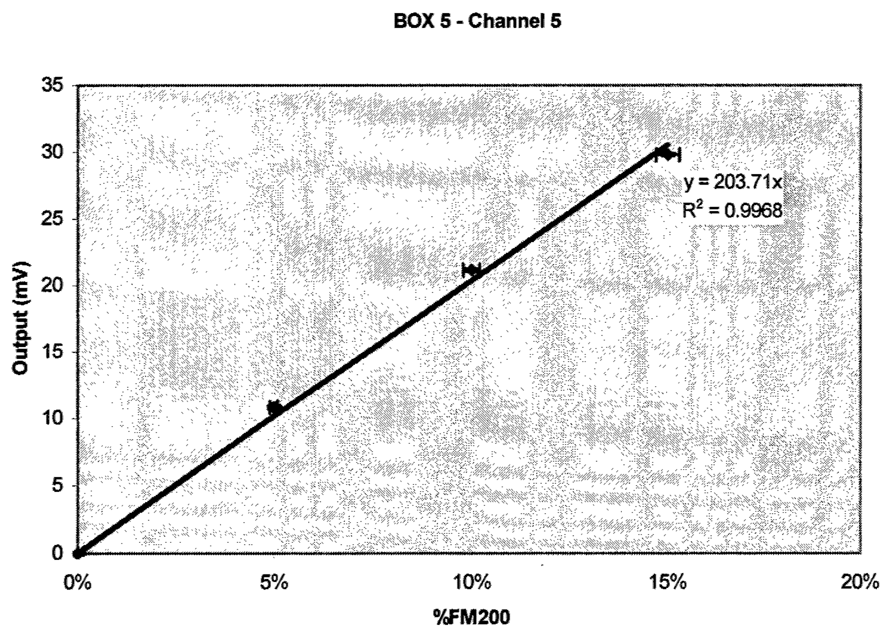


Figure 11(continued) Calibration graphs for Box 5



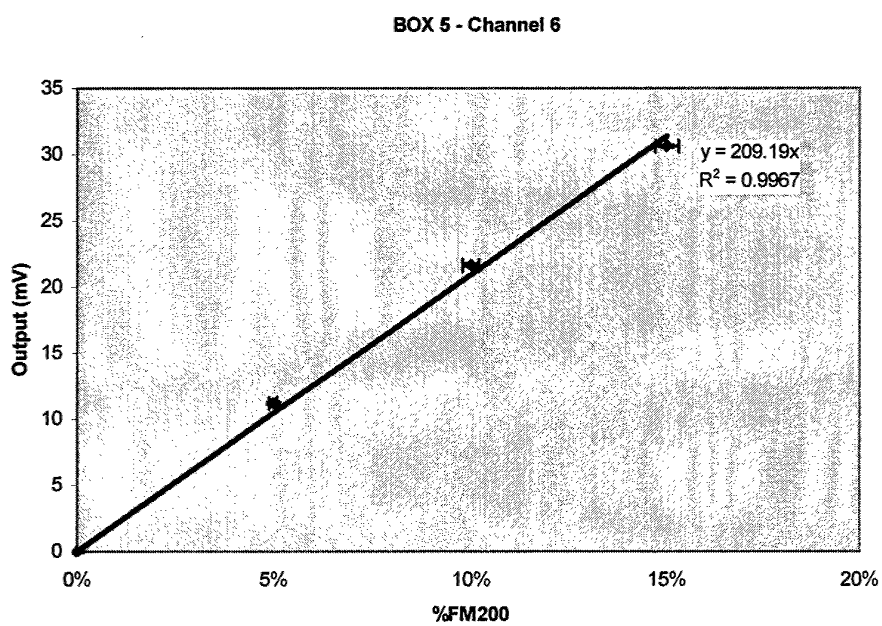


Figure 11(continued) Calibration graphs for Box 5

Calibration equations for each of the calibration graphs were determined by regression analysis, a linear fit chosen because of the linear response of the cell. The equations describing the data in Figs 7-11 are presented in Table 7.

The error bar analysis shows that the calibration equations generally fall within the uncertainties in the measurements of gas volume and analyser output.

Table 7 Calibration equations for each of the gas analysers

		Gas concentration (%)
Box 1	Ch1	Output (mV)/151.9
	Ch2	Output (mV)/172.2
	Ch3	Output (mV)/168.5
	Ch4	Output (mV)/153.8
	Ch5	Output (mV)/172.7
	Ch6	Output (mV)/158.1
Box 2	Ch1	Output (mV)/186.4
	Ch2	Output (mV)/183.1
	Ch3	Output (mV)/171.0
	Ch4	Output (mV)/181.6
	Ch5	Output (mV)/168.8
	Ch6	Output (mV)/171.6
Box 3	Ch1	Output (mV)/197.5
	Ch2	Output (mV)/207.7
	Ch3	Output (mV)/204.6
	Ch4	Output (mV)/209.4
	Ch5	Output (mV)/200.7
	Ch6	Output (mV)/201.8
Box 4	Ch1	Output (mV)/217.2
	Ch2	Output (mV)/202.4
	Ch3	Output (mV)/211.7
	Ch4	Output (mV)/198.9
	Ch5	Output (mV)/215.0
	Ch6	Output (mV)/203.3
Box 5	Ch1	Output (mV)/206.7
	Ch2	Output (mV)/211.3
	Ch3	Output (mV)/202.1
	Ch4	Output (mV)/204.9
	Ch5	Output (mV)/203.7
	Ch6	Output (mV)/209.2

4. Conclusions

1. The methodology used in this paper provides confirmation that the thermal conductivity sensors are essentially linear to 15%FM200

2. The error in gas concentration measurement is within 0.1%FM200 at 5%FM200, 0.1% FM200 at 10%FM200 and 0.2%FM200 at 15%FM200.

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Gas sample preparation and calibration of thermal conductivity gas analysers.

Ian Burch

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19. ABSTRACT This report describes the sample gas preparation technique and calibration procedure for thermal conductivity gas analysers operated by the Platform Sciences Laboratory. The analysers are used to measure gaseous fire suppressant concentrations of extinguishing systems installed in Royal Australian Navy ships. These measurements are undertaken to ensure the systems meet the minimum standards for acceptance into service.						